10

15

20

25

30

## Spectral Analysis System with Moving Objective Lens

#### BACKGROUND OF THE INVENTION

### 1. Field of the Invention

The present invention generally relates to spectral analysis systems that can be used for polymer identification. More particularly, the invention particularly relates to improvements in Raman polymer identification systems to permit effective and rapid identification of darkly colored plastics.

## 2. Description of the Prior Detection Method

Many plastics that should be recycled, particularly in the automotive industry, are black or highly pigmented. Such darkly colored plastics have proven to be the most difficult to identify using existing plastic identification technologies. Due to the strong optical absorption of black plastics, most of the signal needed to perform a spectroscopic identification is absorbed by the sample and thus unavailable for detection. At the same time, absorption may also lead to a significant thermal change such as a rapid heating, melting and even burning of the plastic sample during the identification process. Thus, not only are the signal levels from black plastics very small, but also these weak signals, particularly Raman signals, may be further obscured by large interfering backgrounds due to the thermally induced changes in the plastics including smoking. For example, white plastics can be easily and rapidly identified in 0.1 seconds with a Raman spectrometer, such as that disclosed in International Publication WO 99/01750, using a 1 Watt diode laser power, while black plastics cannot be identified under the same conditions due to laser induced detrimental changes. The power density reaches 5 W/mm<sup>2</sup> when a 1 W laser at a wavelength of 800nm is focused to a 0.5mm diameter focal spot.

In order to avoid laser induces detrimental changes in the plastic, it is necessary to decrease the laser power density on the surface of the black plastic. One way to reduce laser power density is to reduce total laser power that illuminates the surface of the black plastic. But at same time, in order to accumulate enough signal for identification, the signal collection time has to be increased proportionally. Obviously, this is not acceptable for rapid identification. The other way to reduce the power density of the laser is to increase the size of

10

15

20

25

30

the laser spot that illuminates the surface of the plastic, while still maintaining a sufficiently high laser power of 1Watt to allow rapid identification. Experiments have shown that to avoid laser induced detrimental changes in black plastic samples, in the case of 1 Watt total laser power at wavelength 800nm, the size of the laser spot illuminating the surface of a black plastic sample needs to be increased 40 times, to a size that is greater than 3 mm in diameter. As a consequence, the signal acceptance area of the collection fiber bundle and the acceptance area of the spectrograph (slit-height times slit-width) must also be increased 40 times, as illustrated as in Fig 1. It is almost impossible to achieve this from a technical point of view. Enlarging the laser spot size without changing the optical train and components would cause the signal from the sample to overfill the collection fiber bundle and thus decrease the collected signal intensity.

Thus, there exists a need for a quick yet effective way to identify materials such as darkly colored plastics using spectral analysis, particularly Raman spectroscopy.

# **BRIEF SUMMARY OF THE INVENTION**

In order to aid in the understanding of the present invention, it can be stated in essentially summary form that it is directed to a moving objective lens in a traditional system employing spectral analysis such as a Raman polymer identification system. By moving the objective lens, the laser beam can be distributed to a focal plane, while the spectral signal from the moving laser spot can still be collected back to the same point as if the objective lens were stationary. As a result, the average power density of the moving laser spot can be reduced to a point that no light induced detrimental degradation such as undue heating, melting and burning of the plastic sample will occur, while still maintaining the same laser beam power level. At the same time the power level of the spectral signal being returned from the sample is maintained at a level sufficient to make very rapid identification of the character and composition of the sample by an analysis of the Raman or other spectral signal.

In a preferred system, an optical fiber bundle conducts the spectral signal from the sampling optics situated to receive the characteristic spectrum produced from the sample to a spectral analyzer. Even though the lens is moving, the spectral signal returns from the sample to the same point of the entrance end of

10

15

20

30

the fiber bundle as if the lens were stationary. This means the spectral signal is not reduced by the movement of the lens. The terms moving and movement as employed in this application is to be given the broadest possible meaning and include a patterned or random movement of the lens so that the laser energy directed toward the sample is distributed over an area in the focal plane of the objective lens larger than would be achieved were the lens not subjected to movement. Examples of movement that are easily achieved to obtain the desired results include rotation of an eccentrically positioned objective lens, and one- or two-dimensional translational vibration of an objective lens in a plane roughly parallel to the sample surface.

The invention provides a convenient way to solve the problems faced by traditional Raman or other spectral polymer identification methodologies that prevent the rapid and effective identification of darkly colored plastics. The invention can be better understood from the following description when considered in conjunction with the accompanying drawings.

# BRIEF DESCRIPTION OF THE DRAWINGS

Fig-1 shows that when increasing the laser spot size so as to decrease reduce the power density to a manageable lever with a stationary objective lens the Raman signal acceptance area of the fiber bundle needs to be increased.

Fig-2 shows the laser beam and Raman signal path in a traditional Raman Polymer Identification system.

Fig-3 shows the laser beam and Raman signal path in a traditional Raman Polymer Identification system when the objective lens F is off center.

Fig-4 shows the laser beam and Raman signal path in a modified Raman
25 Polymer Identification system with an embodiment of the inventive moving objective lens.

Fig-5 is an exploded perspective view of an identification probe incorporating a spatial filter of the present invention.

Fig-6 shows an objective lens system including a moving objective lens.

Fig-7 shows that tilting movements do not affect the collection efficiency of Raman signal.

10

15

20

25

30

### DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following portion of the specification, taken in conjunction with the drawings, sets forth the preferred embodiments of the present invention. The embodiments of the invention disclosed herein are the best mode contemplated for carrying out the invention in a commercial environment, although it should be understood that various modifications can be accomplished within the parameters of the present invention.

In a traditional Raman Polymer Identification system, such as that disclosed in International Publication WO 9901750, the general signal collection setup can be illustrated as in Fig 2. The objective lens F is placed at (0,0,0). The collimated diode laser beam is focused by the objective lens F at focal point (0,0,f). The Raman signal from the excitation focal point (0,0,f) is re-collimated by the objective lens F and passed through the Holographic Notch Filter (HNF), then collected by lens F1 to fiber bundle at focal point (0,0,L).

When the center of the objective lens F is moved to (a,0,0) as in Fig 3, the focal point of laser beam will be moved to (a,0,f). The Raman signal from the excitation focal point (a,0,f) will be re-collimated by the objective lens F, which is centered at (a,0,0) and passed through the HNF. The Raman signal is then collected by lens F1 at the same point of fiber bundle, (0,0,L). In other words, no matter how the objective lens is moved, as long as it is on the same vertical plane, the Raman signal will always be collected at same point (0,0,L).

When the objective lens F is vibrated on a plane that is perpendicular to the optical axis shown in Figs. 3 & 4, the laser spot that is formed on the sample surface will vibrate on the focal plane synchronously with the objective lens movement in the X and Y directions. With the increase of amplitude of the vibration on the objective lens, the scan area of the focal spot will increase. By adopting random, noisy or complex functions for the amplitude of the objective lens in the X and Y directions, the movement of the laser spot will effectively smear or spread out nearly uniformly over the spot area. As a result, the average power density of the vibrated laser spot can be reduced proportionally until it is lower than the laser induced detrimental change threshold of the sample.

An important key is to recognize that the time it takes for light from the laser to travel from the objective lens to the sample and for the excited Raman

signal to travel back from sample to the objective lens to be re-collimated is less than 1 nano-second (if focal length f < 15 cm). If the vibration frequency is 50 Hz and the vibration amplitude is < 2.5 mm, the mechanical movement of the objective lens F due to the vibration is less than 1 nm during this 1 nano-second time interval. That means the Raman signal will be collected through lens F and F1 and reach the same point of fiber bundle (0,0,L), so the signal will not be weakened due to the mechanical movement of objective lens F. The result of lens vibration with 50 Hz and 2.5 mm amplitude makes the lens spot from 0.5 mm to equivalent laser spot of 5 mm in diameter, which leads to a 100-fold reduction in power density.

This key point of invention is that random, noisy, complex or other movement of the objective lens in the X-Y plane has the same effect as increasing the size of laser spot, yet there is no need to modify the optical system that collects the Raman signal and still maintain the same signal collection efficiency. By applying this invention, the average laser spot size can be easily changed by modifying the amplitude limits on the movement of the objective lens. The present invention has the advantage of averaging the Raman signal that is collected from the test sample over the spot area, which is important when the sample has non-uniformly distributed chemical components. Generally the movement of the objective lens should have a linear velocity component of between about 0.1 to 100 cm/sec.

In a commercial embodiment, the probe 12 is configured to illuminate and collect light scattered from a samples, not shown, that are situated in front of optical window 26 at a front end of nose cone 24 as shown in FIG. 5. Probe 12 includes a housing 14 in the form of a generally cylindrical member 22 and includes a nose cone 24 containing an optical window 26. The optical window 26 can comprise a simple opening through which light can pass, but in a preferred embodiment the optical window 26 comprises a sapphire window mounted within the nose cone 24 to protect the optics within probe 12 from airborne dust and assorted particles. The probe 12 can be easily positioned relative to a sample by means of handle 28 that can constitute a coupling structure for robotic manipulation. A trigger 30 is situated on the handle 28 for easy operation by an operator's index finger. Alternatively, the trigger 30 can be computer controlled.

10

15

20

25

30

A longitudinal rail 32 is fixed to handle 28 or equivalent robotic coupling structure to provide a foundation for the optical components within the probe 12. The generally cylindrical housing member 22 includes a longitudinal slot 16, the edges 18 of which contact opposing edges of the longitudinal rail 32. The housing 22 is completed by back wall 34. In the preferred embodiment, the generally cylindrical housing member 22 has an internal diameter of about 6.0 cm. It is understood, however, that the internal diameter and other dimensions of housing member 22 can vary in accordance with the constraints imposed on the system by its intended use as well as the components to be housed therein. In the preferred embodiment, the housing member 22, nose cone 24, longitudinal rail 32, and back wall 34 are construction of aluminum that has been black anodized. However, a wide variety of metals, copolymers, and composites can be used to construct probe 12 in accordance with the present invention.

The longitudinal rail 32 includes a lower surface 31, an upper surface 33, a rearward end 35, and a forward end 37 as shown in Fig. 5. A plurality of lateral slots 39a through 39g are milled into the upper surface 33 of the longitudinal rail 32 generally perpendicular to the length dimension of the longitudinal rail 32, except slot 39c which is inclined at an angle of about 10°. Pivot pins 38 are fixed in the center of each of the lateral slots 39a and 39c to permit small adjustments in the alignment of the supports fastened therein. Probe 12 employs sampling optics 42 to collect the scattered Raman radiation, discriminating with an extinction ratio of about 10<sup>6</sup> (1 ppm) or better for the Raman-shifted component. Support 46 is fastened in slot 39e to hold lens 36 adjacent the exit end 41 of optical fiber 66 carrying light from a laser source. Support 49 is fastened in slot 39b to hold a band pass filter 48, which controls the wavelength deviation of the source light directed toward the sample through optical window 26. Support 51 is fastened in slot 39a to hold an objective lens system 54 and mirror 50. Supports 46 and 49 also support the ends of baffling tube 47 creating a specific segregated region within the housing 22 between the lens 36 and band pass filter 48.

Support 52 is fastened in inclined slot 39c to hold optical filter 76, which can be an interference or holographic filter and preferably is a long pass filter

15

20

25

30

designed to reflect light having a wavelength equal to or less than the wavelength of the laser source and transmit light having a wavelength longer than the laser source. Support 56 is fastened in slot 39d to hold a lens 74 having a focal length selected to direct the Raman or other characteristic spectral signal passing through the optical filter 76 on to the entrance end 53 of spatial filter 55. Support 58 is fastened in slot 39e to hold the entrance end 53 of spatial filter 55. The entrance end 53 of the spatial filter 55 includes an aperture 65 that is generally round and preferably has an area of about 1mm² or less. Support 40 is fastened in slot 39g to hold the exit end 57 of spatial filter 55 that also holds the entrance end of optical fiber bundle 62 that carries the characteristic Raman or other spectral signal produced from a sample through the fiber-optic bundle 62 to appropriate instruments capable for evaluating the spectral signal. The specific structure of the preferred embodiment of the spatial filter 55 is disclosed in copending U.S. Patent application SN 09/447,878 filed November 23, 1999, which is hereby incorporated by reference.

A convenient method of achieving the desired movement of the objective lens 60 is to mount the lens in a lens holder 59 as shown in Fig 6 so that the lens center C is displaced from the optical axis Z. The objective lens 60 is thus eccentrically mounted with respect to the optical axis Z of the probe 12 and then simply rotated by motor 61. This rotation causes the focal point of the lens 60 to rotate in the plane of the lens holder 59, which is generally parallel to the surface of the sample, about the optical axis Z of the probe 12, describing a circle that has radius equal to the eccentricity of the lens mount 59. In the preferred embodiment, the rotating objective lens 60 is mounted adjacent to or in the nose 24 of the probe 12. The lens mount 59 is rotated by the electrical motor 61 upon depression of the trigger 30, which can also initiate the emission from the laser source through electrical cables 69 and 70. The rotation of the lens holder 59 has the effect of causing the laser spot to scribe a circle on the test sample.

The amount of lens displacement from the optical axis and the speed of rotation can be selected so that the power density of the excitation laser beam is reduced until it is lower than the laser induces detrimental change threshold of the sample. For example, a lens can be mounted in a lens holder so that the lens

center is displaced from the optical axis by about 1.5 mm, which causes the focal point of the excitation laser beam to scribe a circle having a diameter of about 3.0 mm as shown in Fig. 7. When this lens is rotated, the power density of the excitation laser beam is distributed over a circle described by the rotation of the focal spot. In the limit of a rotation rate that is faster than thermal diffusion, the steady-state power density falls by a factor of A/A', where A is the area of the static focal spot and A' is the area of the ring illuminated by the rotating spot. This limiting ratio is described by the formula r/4R, where r is the radius of the focal spot and R is the radius of the ring described by the rotation of the focal spot. The eccentricity of the lens mounting can be from about 0.05 to 1.0 cm while the movement of the lens holder varies between about 0.1 and 100 rev/sec. Correspondingly, when the lens holder is vibrated at a frequency of from about 0.1 to 100 Hz, the amplitude of the vibration can be varied between about 1.0 and 0.01 cm.

It should be noticed that any tilt movement during the objective lens vibration will not affect the collecting efficiency of Raman or other spectral signal illustrated In Fig 7. In other words, there is no strict requirement for the accuracy of the objective lens movement, although significant translational movements of the objective lens in the Z direction could affect the collimation of the returning spectral signal by the objective lens 60.

Although the invention has been described in detail with reference to a preferred embodiment, variations and modifications exist within the scope and spirit of the invention as described and defined in the following claims.